S. L. Peterson

The Installation of an Arsem Electric Vacuum Furnace

THE INSTALLATION OF AN ARSEM ELECTRIC VACUUM FURNACE AND THE DETERMINATION OF MELTING POINTS OF DEOXIDATION SLAGS

BY

SIDNEY LE ROY PETERSON

THESIS

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THIS IS TO CERTIFY THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

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ENTITLED THE INSTALLATION OF AN ARSEM ELECTRIC VACUUM FURNACE

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D. F. McFarland

Instructor in Charge

APPROVED:

HEAD OF DEPARTMENT OF CHEMISTRY

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TABLE OF CONTENTS

THE DETERMINATION OF MELTING POINTS OF DEOXIDATION SLAGS

THE INSTALLATION OF AN ARSEM ELECTRIC VACUUM FURNACE

ILLUSTRATIONS, TABLES AND CURVES

ILLUSTRATIONS

TABLES AND CURVES

THE DETERMINATION OF MELTING POINTS OF DEOXIDATION SLAGS

I INTRODUCTION AND PURPOSE OF INVESTIGATION

June 25th, 1918, a letter was received from Dr. H. M. Howe, Chairman, Engineering Division, and Dr. Bradley Stoughton, Chairman, Section on Metallurgy of the National Research Council, in which was discussed a problem of great importance for winning the war. The contents of the letter was substantially as follows:

"This country has no sufficient supply of minerals rich enough in manganese to supply the steel industry with the ferro-manganese which it now uses. It is believed that manganese can be replaced in very large part by other deoxidizing agents combined in suitable ratios such as silicon, aluminium, titanium and possibly calcium and magnesium. It is also believed that an essential to replacing manganese with other deoxidizing agents is that the resultant oxides shall be in such ratio as to form a fusible compound, which will rise to the surface, and the immediate objective is to learn what combinations of oxides are thus fusible at temperatures far enough below the melting point of the steel to insure their rapid separation by gravity. This separation requires that the oxides shall be very liquid, so that the individual particles may coalesce and rise to the surface".

The Committee on Substitute Deoxidizers appointed by the Bureau of Standards with collaboration of the National Research Council and the Geophysical Laboratory chose the University of Illinois as one of a group of eleven cooperating laboratories to

determine the melting points of new deoxidizers and new deoxidizing alloys, the discovery of which might lead to providing war-time substitutes for manganese or economize in the use of that element for steel deoxidation.

In June, 1918, a conference was held between Dr. J. R. Cain, Chairman, Committee on Substitute Deoxidizers and Dr. H. M. Howe, Chairman, Engineering Division of the National Research Council, at which the following ideas were discussed:*

"(1) One of the most desirable properties of a deoxidizer for metals is that of forming a deoxidation slag, fusible enough, and fluid enough to separate readily from the metal.

"(2) New deoxidizers could be selected by this criterion.

"(3) New deoxidizing alloys could be discovered by making from oxides of the elements in the alloy, synthetic slags such as would probably result upon its use for deoxidation, and test such slags for melting point, fusibility and viscosity."

In view of the specialized experience of Dr. R. J. Sosman of the Geophysical Laboratory on silicate research, it was thought that he should be able to advise particularly as to literature. He stated that practically nothing was available on viscosities and very little that would throw any light on fusibility and melting points of the special slag mixtures involved in this proposed investigation. It was decided that preliminary determinations should be limited to binary and ternary mixtures in so far as possible limiting the binary mixtures to pairs of acidic and basic oxides (e.g. silicates, aluminates, and titanates) combined with a

* Letter from J. R. Cain

basic oxide and to apply the same rule to tern ary mixtures in so far as possible. Dr. Sosman suggested selecting the ter nary mixtures from the interior of a triangular coordinate diagram.

Synthetic oxide mixtures in various proportions of elements such as silicon, aluminium, titanium, manganese and vanadium which have been known to be useful singly in deoxidizing steel were selected.

Dr. J. R. Cain suggested that the oxide mixtures be limited to those melting at 1500° C or below, since no thers would be useful in steel metallurgy. Also as automatically accomplishing this result, he suggested the use of commercially pure iron crucibles (melting point about 1540° C) used in an inert atmosphere as containers for the melts. If the crucible melted before the mixtures, that mixture would be automatically excluded.

All of the above suggestions were adopted at this conference. A series of 120 binary and ternary mixtures of oxides of manganese, aluminium, silicon and titanium were made up and distributed to the various cooperating laboratories.

Twelve samples were received by this laboratory from Dr. J. R. Cain, Chairman, Committee on Substitute Deoxidizers, National Research Council, consisting of oxide mixtures of manganese and silica varying by 5 percent (weight) steps from 40 to 95 percent silica and 60 to 5 percent manganese as well as a standard mixture composed of 55 percent TiO_2, 20 percent MnO, 25 percent Al_2O_3. The melting point of the standard mixture as determined by the Bureau of Standards was 1370° C.

II FACTORS AND CONDITIONS

The factors and conditions that affect the observed values of melting points of these mixtures are the following: chemical composition, size of particles, shape and position of mould, time and rate of heating, and nature of surroundings. In the determination of the melting points of the various mixtures, the above factors must be kept constant, the only factor being varied is the composition.

In the strictest sense, the term melting point is applied to the temperature at which the solid and liquid phases of a pure crystalline substance can remain in equilibrium. In the case of a mixture of oxides, the term melting point is not a definite temperature; the change from the solid condition to one in which the material will flow is gradual over a temperature and time interval.

In these determinations, the sample was moulded in the form of a cylinder, the beginning of the deformation marks the first stage of melting; the second stage begins when the material has fused into a lump or ball; the third stage begins when the lump has flattened out and is fluid (5).

The temperature at which a marked and distinct flow of the sample began was taken as the melting point in these determinations

III METHOD

The finely ground silica and manganese carbonate were thoroughly mixed and moistened with water and moulded into cylinders 1

centimeter in length. The cylinders of slag mixtures were air dried for two or more days before using.

The melting points were determined by direct observation in an Arsem electric vacuum furnace. The cylinders of slag mixtures were placed in "Armco" iron crucibles furnished by Committee on Substitute Deoxidizers. The crucibles were placed in the alumina crucible 1-1/4 inches in diameter by 4 inches in length, supported on a graphite holder. The crucibles were placed slightly below the hottest part of the furnace so that any change in shape of the material was visible through the observation window of the Arsem furnace . The temperature was raised gradually until evidence of softening or melting appeared. The melting point was observed with a Leeds Northrup Optical pyrometer at the temperature at which the sample would flow. The gaseous pressure in furnace was about .2 centimeter or less throughout the work. However, no accurate pressure measurements could be made because of the limitations of the manometer used. At about 800° C, carbon dioxide from the manganese carbonate was evolved, leaving the manganese in the form of MnO.

IV RESULTS

Standard Mixture

Sample No.	Composition	Melting Point °C
89	25% Al_2O_3, 20% MnO, 55% TiO_2	1384
89	"	1379
89	"	1383
	Average	1382

Melting Point as determined by Bureau of Standards - 1370° C

Sample No.	Composition		Melting Point
40	10% MnO	90% SiO_2	above iron (1602°)
41	15%	85%	above iron
42	20%	80%	above iron
43	25%	75%	above iron
44	30%	70%	above iron
45	35%	65%	
46	40%	60%	
47	45%	55%	
48	50%	50%	
49	55%	45%	
50	60%	40%	

The determination of the melting points of samples 45-50 was impossible owing to the breaking down of the heating element and the inability to replace it within the time allowed for completion of the thesis.

V CONCLUSION

The slag mixtures of the following composition

Sample No.		
40	10% MnO	90% SiO_2
41	15%	85%
42	20%	80%
43	25%	75%
44	30%	70%

would not be useful in steel metallurgy because their melting points are above that of molten iron or steel. No opinion can be ventured as to the utility of the slag mixtures whose melting points were not determined.

THE INSTALLATION OF AN ARSEM ELECTRIC VACUUM FURNACE

I GENERAL

In order to carry out the determinations of melting points of the slag mixtures, it was necessary to select a furnace appropriate for the problem. The selection of a graphite resistance vacuum furnace of the Arsem type was based upon its points of merit such as: the lasting operation of the resistor, the clarity of the atmosphere, the rapidity of action and excellence of control and the good black body conditions obtainable, the presence of a non-oxidizing and practically non-reducing atmosphere which would insure the oxide constituents of the slag being present in the state of oxidation usual in such slags.

The installation consists of a 15 kilowatt Arsem vertical electric vacuum furnace, a Perkins oil vacuum pump, a 20 kilowatt oil cooled Peerless transformer, a field rheostat (5 ohms, 60 amperes, 440 volts) and mercury manometer.

Other essential accessories are a Leeds and Northrup optical pyrometer, a 15 kilowatt wattmeter and other electrical measuring instruments

II CONSTRUCTION

The general appearance and details of construction of this type of furnace is shown in Illustrations No. I, II and III.

The vacuum furnace (2)(3) is a special type of resistance furnace enclosed in a vacuum chamber. The vacuum chamber is an air tight vessel of gun metal, the surface being machined and turned to close the pores. Tubular electrodes enter the cover

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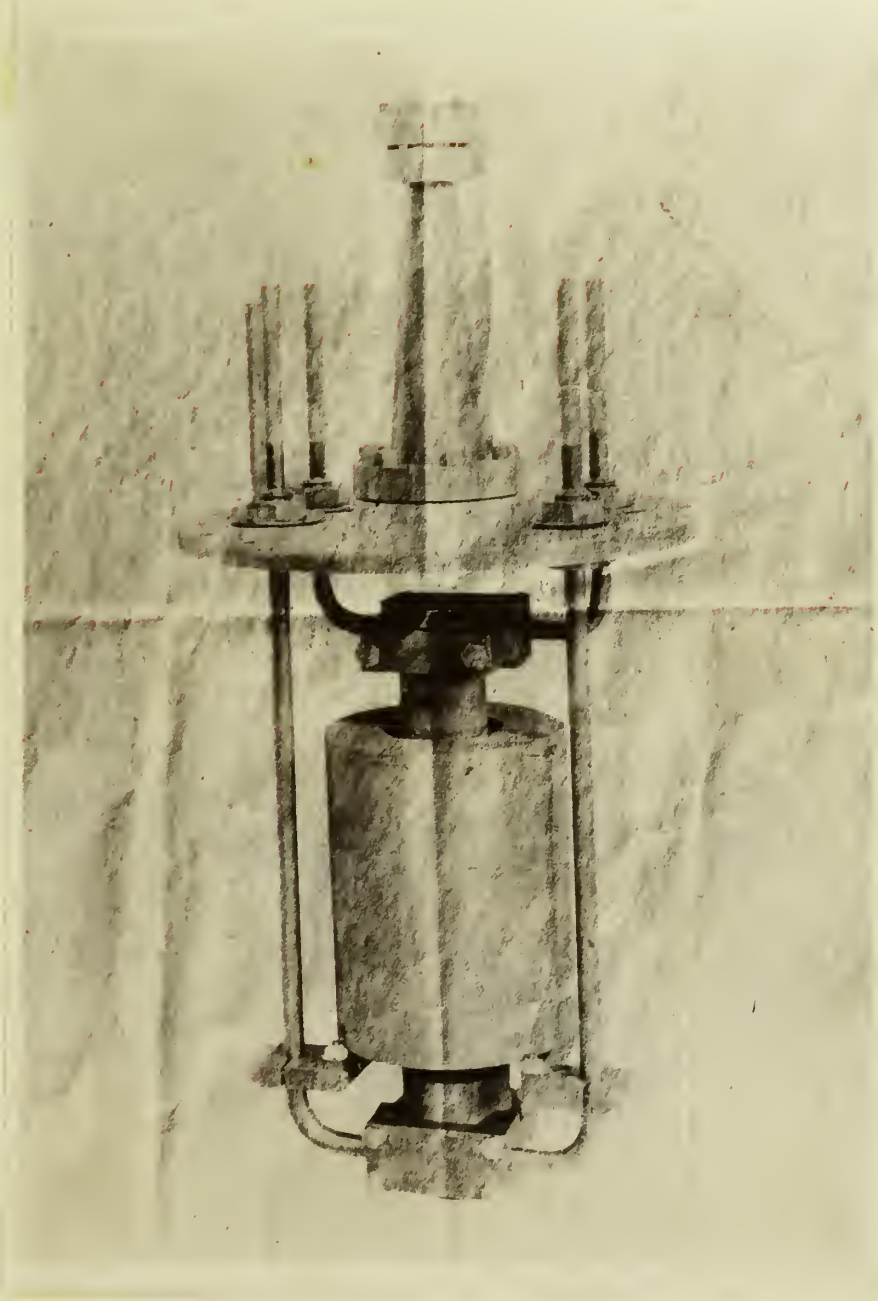


and are provided with clamps which hold the heater in place and make contact between it and the electrodes. All joints are made tight by lead gaskets and the electrode joints have to be made so that they are air-tight, electrically insulated, and not liable to deterioration by heat. The radiation screen which surrounds the heater to diminish the radiation loss is also held in place by supports, attached to the electrodes. The details of construction will be better understood by reference to Illustration No. II.

The chamber (A) and cover (B) are castings which are turned true at the joint. A lead gasket (C) 1/16" thick, forms an air-tight joint when the cover is fastened down with the cap-screws (D). The first time the furnace is assembled, the tightening of the screws forces the lead into the annular grooves of the chamber and cover. The chamber has four legs to permit flow of water underneath. The tube (J) through which the air is exhausted is soldered into the cover.

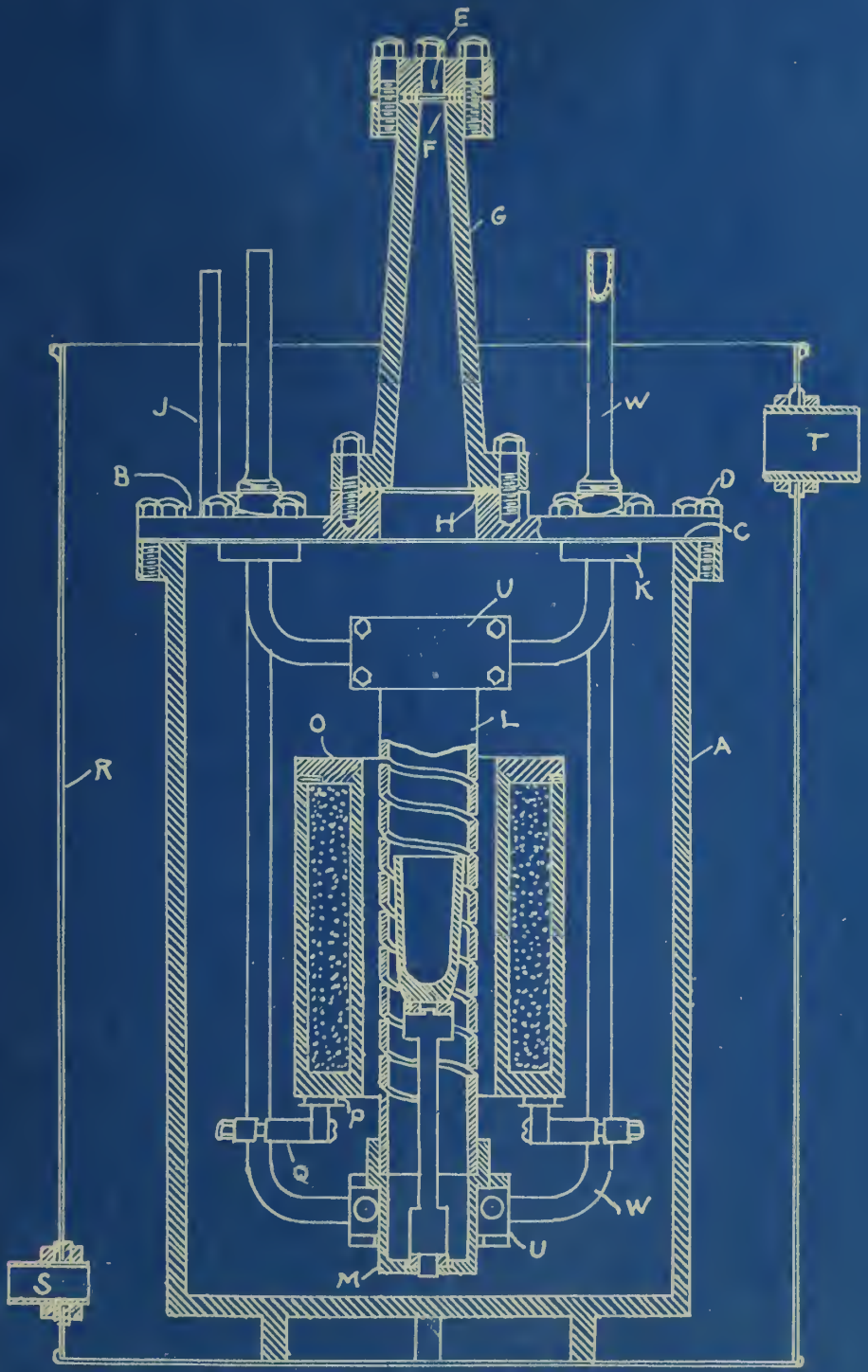
The window (E) is a disc of clear white mica about .005 inches think, clamped between two lead washers (F), by means of a brass cap and four cap-screws. The brass surfaces touching the lead washers have annular grooves into which the lead is forced by pressure. The window tube (G) is of bronze, and is fastened to the cover by six cap-screws, the joint being made tight by a lead washer (H).

The electrodes (W) are formed of brass tubing bent into shape. The threaded brass bushing (KK), which constitutes parts of the electrode joints, are soldered to the electrode tubes. The heater

clamps (VV) are of copper.

The heater (L) is a helix of artificial graphite which has a negative temperature coefficient. The resistance is approximately .35 ohm. Thus, if 60 volts are impressed directly on the terminals of the furnace, 250 amperes will flow. This is the maximum voltage which should be impressed upon the terminals. Sixty volts will heat the furnace to an extremely high temperature (as can be seen from the temperature vs. energy curve, Curve No. I) and should be used only very rarely for short periods of time. The heater is made by first cutting a helical groove of the desired pitch and depth, and then cutting out the core by means of a pipe provided with teeth. Care has to be exercised to obtain a perfectly uniform cross section. Its dimensions and the number of turns are so related that at the highest temperature reached the potential difference across the heater shall not be more than 50 volts in order to avoid excess Edison effect, and the tendency to arc across to the screen. The graphite cup (M) holds the lava insulating ring on which the crucible support rests. The proximity of this ring to the cold bottom of the furnace prevents it from fusing or being made conducting by heat.

The radiation screen (O) is a double-walled cylindrical box of Acheson graphite, filled with graphite powder. Its purpose is to minimize loss of heat by radiation, thereby increasing the efficiency of the furnace. It was found that with the use of the screen, the melting point of platinum could be reached with but one-fourth of the energy necessary without it. The screen is



SECTIONAL ELEVATION OF ELECTRIC VACUUM FURNACE.

supported by the copper arm (Q) from which it is insulated by the lava buttons (P).

The water jacket (R) is a galvanized iron tank provided with an inlet (S) and an outlet (T). The electrodes are hollow so that water may be passed through them in order to keep them from deteriorating by heat.

The overall height of the assembled furnace is approximately 27-1/2 inches, including the window tube. The water jacket is 15 inches in diameter and 31 inches high.

III CONTROL AND OPERATION

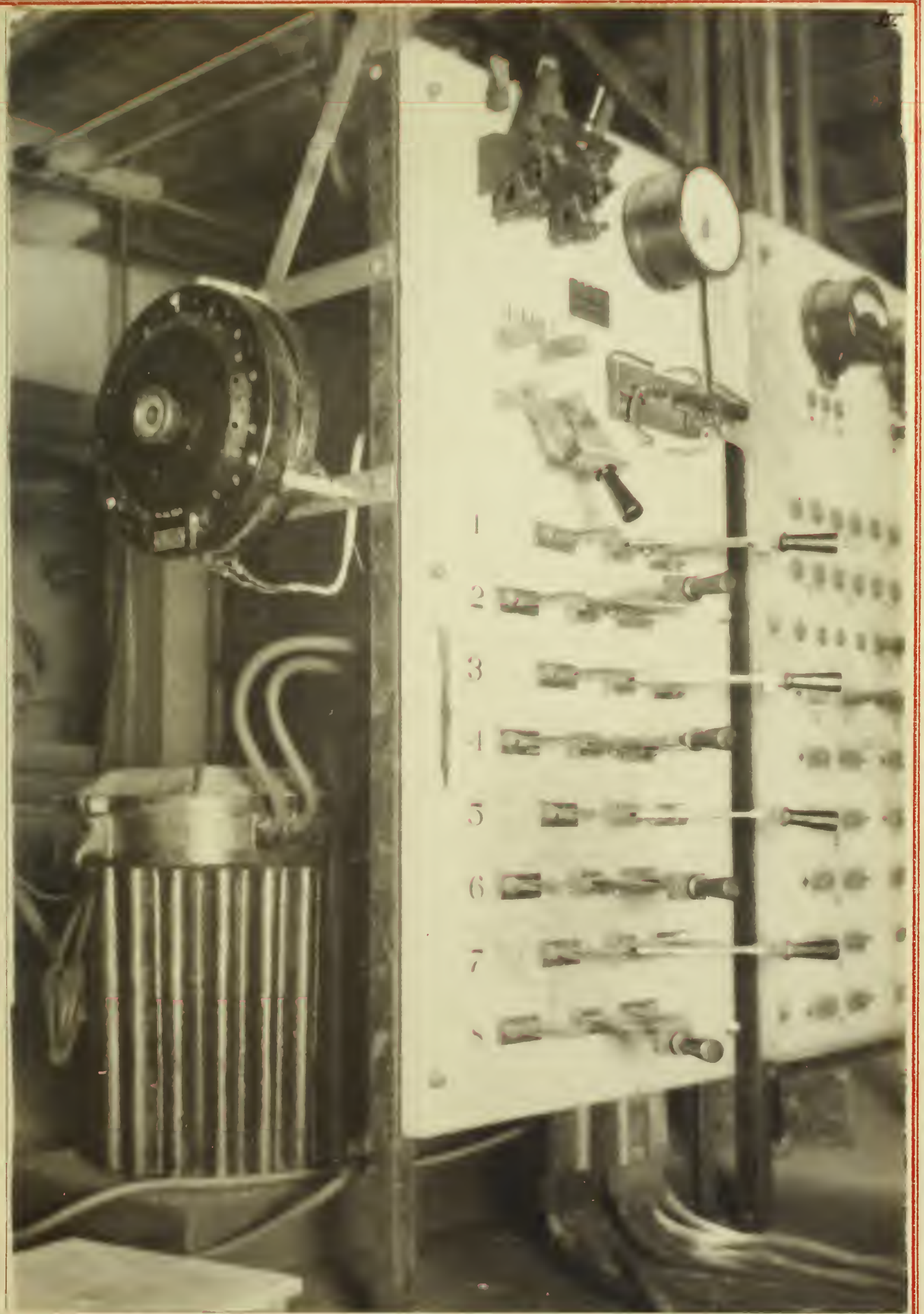
The range of voltage required for this furnace is 60 volts maximum and 15 volts minimum and is usually obtained by using a 37-1/2 volt transformer and a standard induction regulator designed for 100 percent boost and buck, type IRS, 60 cycle, 6-1/4 KVA to be used on 40 volt transformer connection.

In designing the installations where field control as a means of regulating the voltage on the furnace is not permissible, several other means of securing variable voltages are available:

1. A transformer with a series of taps giving different voltage.

2. An ordinary transformer used in connection with an induction regulator.

3. A "flux shunt" or pipe thawing transformer. Owing to the fact a 30 kilowatt Peerless transformer was available, it was decided a 37-1/2 volt transformer as recommended by the General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Electric Company was not needed, and that the Peerles transformer could be used for obtaining the proper voltage. Because of the high cost of induction regulators, this form of regulator was found out of the question. It was decided to use a field rheostat (5 ohms, 60 amperes, 440 volts) in order to obtain regulation between the various steps of the transformer.

The Peerless transformer has a capacity of 20 kilowatts and is oil cooled. The primary is designed for alternating current at 60 cycles per second, 440 volts, with a tap from the center of the coil so that it can be operated at 220 volts by connecting the two halves in parallel. The secondary consists of four separate coils two to deliver 10 volts each and two to deliver 20 volts each. The two 10 volt coils develop exactly equal voltages so that they may be used in parallel.

The voltage of these two coils in series equals that developed by the 20 volt coils and thus allows them to be connected in series, parallel with said 20 volt coils, which likewise generate equal voltages.

Terminals of the coils are brought out as cables, the secondary three feet long and those for the primary six feet long. The transformer is capable of operating continuously at 25% above its rated capacity without injury.

The terminals of the secondary are connected to a switchboard having eight switches whereby it is made possible by throwing the switches to the right or left or having them open to obtain steps of 10, 20, 30, 40, 50, and 60 volts respectively. See Table No.

III. This is exactly the range required for a 15 kilowatt Arsem electric vacuum furnace. In order to obtain regulation between the above mentioned steps, a field rheostat (5 ohms, 60 amperes, 440 volts) is placed in the primary side of the transformer, and by introducing resistance, the current may be lowered thereby resulting in the decrease of the voltage. By introducing approximately two ohms resistance, the voltage may be decreased 10 volts thereby making it possible to have complete regulation of the voltage.

A shunt switch was placed in the line so that if the use of the rheostat was not necessary, the rheostat may be cut out of the primary line by closing the switch.

A 15 kilowatt wattmeter connected in the secondary line is very helpful in that it indicates the energy being consumed by the furnace. See Curve No. I. By reference to this calibration curve, the temperature may be obtained fairly accurately.

The crucible is placed on the support and centered so that it does not touch the heater, the window tube is then fastened down, the washer and contact surfaces having been most carefully cleaned.

When a good vacuum has been established, the current is switched on and the water allowed to flow through the jacket so that it comes out barely warm, the water level being above the window joint. Water is also passed through the electrode tubes, the out flow being allowed to play against the side of the window tube.

In most cases, the current should be raised gradually, so that the behavior of the heated substance can be followed. It is also

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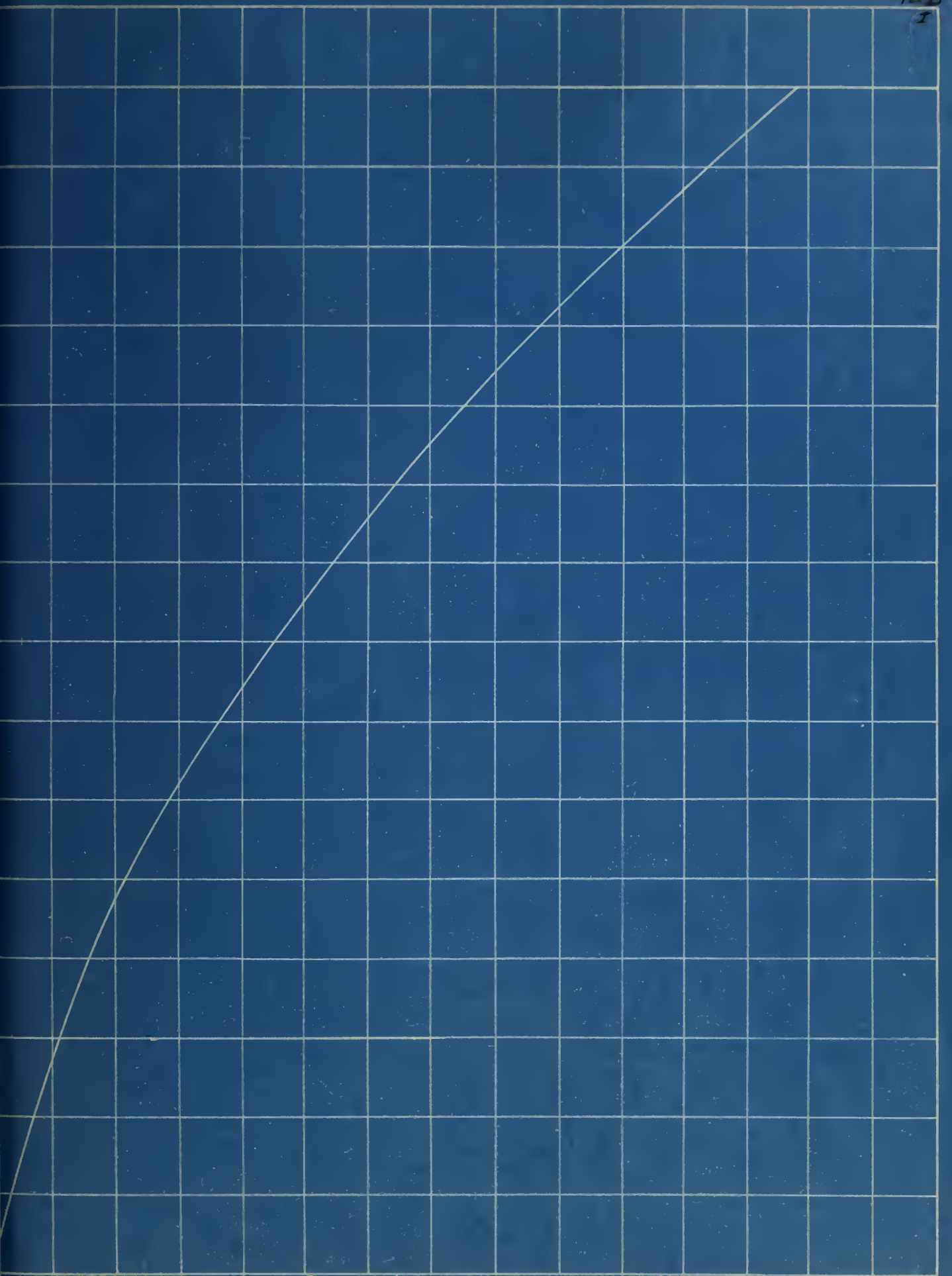

ELECTRIC FURNACE CALIBRATION CURVE

OUTLINE of SWITCHBOARD CONNECTIONS

10 Volts 6-⅔ K.W.		20 Volts 20 K.W.		30 Volts 20 K.W.	
Switch #1	R	Switch #1	R	Switch #1	L
2	R	2	L	2	R
3	R	3	L	3	L
4	R	4	R	4	R
5	Out	5	R	5	R
6	Out	6	R	6	L
7	Out	7	R	7	R
8	Out	8	R	8	L

40 Volts 13⅓ K.W.		50 Volts 16⅔ K.W.		60 Volts 20 K.W.	
Switch #1	Out	1	Out	1	R
2	Out	2	Out	2	L
3	Out	3	R	3	L
4	Out	4	L	4	L
5	R	5	L	5	L
6	L	6	L	6	L
7	L	7	L	7	L
8	R	8	R	8	R

Pull High Tension Switch before Changing Low Tension Switches

R-means Switch to Right.

L-means Switch to Left.

important that the rate of heating be taken into consideration as is later explained.

The range of temperature extends to 3100° C using a maximum of 15 K. W., but most experiments do not require a higher temperature than 2500° C, which can be obtained with 10 K. W., using the 50 volt step of the transformer.

The substance or article being heated is observed through the mica window, cobalt glass being used to protect the eyes at temperatures above 1100° C. At 2500° C and above, three or more thicknesses of cobalt glass are necessary to make it possible to distinguish the outlines of the substance because of the intensity of the light.

When the desired result has been attained, the circuit is opened; from one to three hours are allowed for cooling, according to the temperature reached, then the water is let out of the jacket, air is admitted to the furnace, the window tube is unfastened and the crucible removed.

IV CALIBRATION OF FURNACE

The accurate control of the temperature is made possible by a calibration of the furnace, to determine the relation of the temperature to the energy. The temperature can be quickly brought to any desired point, as determined by the calibration curve, and maintained constant for long periods, while the behavior of the article being heated may be observed through the mica window at the top.

Temperature calibration curves for this furnace are plotted by means of the equation $(y - 20)^2 = ax$ (2) in which y is the centigrade temperature of a crucible in the furnace and x is the corresponding energy in kilowatts. The constants n and a are obtained by determining the energy necessary to maintain temperature equilibrium at the melting points of copper and platinum.

Greater accuracy in temperature observations can be obtained with a Leeds and Northrup optical pyrometer of the Morse, Holborn Kurlbaum type, which can be easily calibrated by comparing it with an electric resistance pyrometer or by the melting points of standard substances such as gold, copper and platinum.

V USES

This type of furnace is especially useful for research work and small scale experiments that can be performed in crucibles 1-1/2 inches in diameter and 4 inches in height.

The following are some of the chief uses(2)(3):

Preparation of metals, alloys, carbides, silicides, and other compounds.

Determination of melting points of metals, alloys, glazes, slags, refractories, etc., by an optical pyrometer, or by reference to the furnace calibration curve.

Distillation of refractory substances for separation and purification.

Study of equilibrium in reactions depending upon the pressure of the gaseous phase.

Many reactions can be studied quantitatively with accurately weighed quantities.

Metals obtainable only in the form of a powder containing impurities can be brought into a pure compact state by fusion in vacuuo, as the non-volatile impurities form a separate layer and the volatile ones can pass off.

Other substances which cannot be heated in air without oxidizing, can be fused in vacuuo without difficulty, unless they vaporize before their melting points. Aluminium oxide, platinum and other refractory materials have been fused while magnesium oxide, iron, silicon and aluminium have been vaporized.

Chemical reactions, synthesis and reductions can be carried out with great facility under direct observation.

Gas producing reactions can be followed closely by means of the manometer, which shows the temperature at which the reaction begins and the point at which the reaction is ended.

TABLE OF FURNACE DATA
for
SMALL VERTICAL ELECTRIC VACUUM FURNACE

Maximum Kilowatts	15
Maximum Current Amperes	250
Maximum Voltage	60
Maximum Temperature	3100 deg.
Crucible Volume	5 cu. in.
Crucible Height	4 in.
Crucible Section	1 1/4 in. diam.
Height of Furnace	21 in.
Base of Furnace	15 in. diam.

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